



# FLAME- AND SMOKE-RETARDANT POLYMER SYSTEMS

Fourth Interim Report

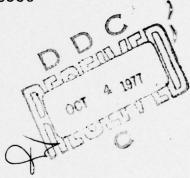
Issued: 15 September 1977

Period Covered: February 1977 - August 1977

Prepared Under Contract: N00024-76-C-5336

For

Department of the Navy Naval Sea Systems Command Washington, DC 20362



Leo Parts, Robert D. Myers and Norman F. May

# MONSANTO RESEARCH CORPORATION

A SUBSIDIARY OF MONSANTO COMPANY



DAYTON LABORATORY

DAYTON, OHIO 45407

A STATUTE PRINCES

## FLAME- AND SMOKE-RETARDANT POLYMER SYSTEMS

Fourth Interim Report

Issued: 15 September 1977

Period Covered: February 1977 - August 1977

For

Department of the Navy Naval Sea Systems Command Washington, DC 20362



Leo Parts, Robert D. Myers and Norman F. May

MONSANTO RESEARCH CORPORATION DAYTON LABORATORY DAYTON, OHIO 45407

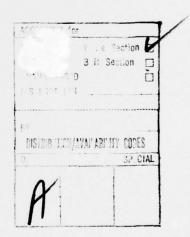
/ See 1473)

## ABSTRACT

The effort directed to the enhancement of the fire performance characteristics of molded polyvinyl chloride was continued. The effects of a latex-based intumescent paint and a mastic coating on the formation of smoke and volatile combustion products were determined. Also, flame propagation measurements were conducted with these and with other, previously prepared experimental compositions.

Among the protective coatings evaluated, an alkyd-based intumescent paint exhibited best performance with regard to smoke and CO formation. This coating also contributed least to the  $\mathrm{NO}_{\mathrm{X}}$  content of the combustion chamber atmosphere.

Tensile strength, dielectric constant, dissipation factor and volume resistivity data are reported for the experimental fireand smoke-retardant PVC compositions.



# TABLE OF CONTENTS

			Page
1.	INTRO	DUCTION	1
2.	EXPER	IMENTAL	1
	2.1	Materials	1
	2.2	Test Methods for Smoke, Gaseous Combustion Products, and Flame Propagation	1
	2.3	Tensile Strength and Elongation Measurements	2
	2.4	Electrical Measurements	2
3.	RESUL	TS AND DISCUSSION	2
	3.1	Formation of Smoke	2
	3.2	Formation of Gaseous Combustion Products	3
	3.3	Flame Propagation	23
	3.4	Tensile Strength and Elongation	26
	3.5	Electrical Properties	27
4.	ACKNO	WLEDGMENTS	27
5.	REFER	ENCES	27
APPE	NDIX		29
DIST	RIBUTI	ON LIST	31
חח פ	וור אפר	73	32

# LIST OF TABLES

Table		Page
I	Smoke Optical Data for PVC Compositions	4
II	Sample Mass Data	8
III	Maximum CO Concentration Data for PVC Compositions	9
IV	$\begin{array}{lll} {\tt Maximum~CO_2~Concentration~Data~for~PVC} \\ {\tt Compositions} \end{array}$	13
V	$\begin{array}{ll} {\tt Maximum~NO_X~Concentration~Data~for~PVC} \\ {\tt Compositions} \end{array}$	14
VI	Maximum HCl Concentration Data for PVC Compositions	17
VII	Maximum Hydrocarbons Concentration Data for PVC Compositions	19
VIII	Flame Propagation Measurement Results	24
IX	Tensile Strength and Elongation Data for Some PVC Compositions	26
X	Electrical Properties	27
XI	Experimental Flame Propagation Data	30

# LIST OF ILLUSTRATIONS

Figure		Page
1	Maximum Specific Smoke Optical Densities of FSP Compositions	5
2	The Effect of Intumescent Coatings on Smoke Formation from 1/16 in. Thick Samples of FSP-1	6
3	The Effect of Intumescent Coatings on Smoke Formation from 1/16 in. Thick Samples of FSP-2	6
4	Maximum Smoke Optical Densities Generated from Intumescent Coatings, Applied Onto Aluminum Foil	7
5	The Effect of Intumescent Coatings on Maximum CO Concentrations Developed from 1/16 in. Thick Samples of FSP-1	10
6	The Effect of Intumescent Coatings on Maximum CO Concentrations Developed from 1/16 in. Thick Samples of FSP-2	10
7	The Effect of Intumescent Coatings on Maximum CO Concentrations Developed from 1/4 in. Thick Samples of FSP-1	12
8	The Effect of Intumescent Coatings on Maximum CO Concentrations Developed from 1/4 in. Thick Samples of FSP-2	12
9	The Effect of Intumescent Coatings and Maximum $\mathrm{NO}_{\mathrm{X}}$ Concentrations Developed from 1/16 in. Thick Samples of FSP-1	15
10	The Effect of Intumescent Coatings on Maximum $\mathrm{NO}_{\mathbf{X}}$ Concentrations Developed from 1/16 in. Thick Samples of FSP-2	15
11	Maximum $\mathrm{NO}_{\mathbf{X}}$ Concentrations Generated from Intumescent Coatings that Have Been Applied onto Aluminum Foil	16
12	The Effect of Intumescent Coatings on Maximum HCl Concentrations Developed from 1/16 in. Thick Samples of FSP-1	18
13	The Effect of Intumescent Coatings on Maximum HCl Concentrations Developed from 1/4 in. Thick	18

# LIST OF ILLUSTRATIONS (cont'd)

Figure		Page
14	The Effect of Intumescent Coatings on Maximum Hydrocarbons Concentrations Developed from 1/16 in. Thick Samples of FSP-1	20
15	The Effect of Intumescent Coatings on Maximum Hydrocarbons Concentrations Developed from 1/16 in. Thick Samples of FSP-2	20
16	Maximum Hydrocarbons Concentrations Generated from Intumescent Coatings that Have Been Applied Onto Aluminum Foil	21
17	The Effect of Intumescent Coatings on Maximum Hydrocarbons Concentrations Developed from 1/4 in. Thick Samples of FSP-1	22
18	The Effect of Intumescent Coatings on Maximum Hydrocarbons Concentrations Developed from 1/4 in. Thick Samples of FSP-2	22
19	Flame Propagation by FSP-1 Compositions	25
20	Flame Propagation by FSP-2 Compositions	25

## 1. INTRODUCTION

Means for enhancing the fire safety of two polymeric materials, plasticized polyvinyl chloride (PVC) and Neoprene, are investigated in this program. As reported earlier (Ref. 1), cupric and ferric acetylacetonates were found to reduce smoke formation from 1/16 in. thick samples of PVC, in terms of the optical density, by approximately 55%. Quantitative data have now been obtained about smoke optical densities and gaseous products formed during the combustion of thick (0.25 in.) samples of fire- and smoke-retardant (FSR) compositions.

In an attempt to enhance the fire performance of the FSR compositions, they have been coated with intumescent compositions. Data regarding the effects of two intumescent coatings on the formation of smoke and gaseous combustion products were reported earlier (Ref. 2). Similar information is presented here regarding another type of intumescent coating and a mastic.

The results of flame propagation, electrical and physical measurements with the experimental FSR compositions are also presented in this report.

## 2. EXPERIMENTAL

#### 2.1 MATERIALS

The compositions FSP-1 and FSP-2, containing ferric and cupric acetylacetonate, respectively, and the sample fabrication procedure have been reported previously (Ref. 1 and 2).

A latex-type intumescent paint (No. 330 by Ocean Chemicals, Inc.) and a mastic (No.  $3^{4}$  by the same manufacturer) were evaluated as the two additional protective coatings. The white intumescent paint was applied with a brush at a coverage of  $^{\circ}200$  square feet per gallon; the thickness of the coating after one week of drying at room temperature was approximately 12 mils. The mastic was applied with a spatula at a thickness of 0.12 in. These samples were also allowed to dry at room temperature for at least one week before they were evaluated.

# 2.2 TEST METHODS FOR SMOKE, GASEOUS COMBUSTION PRODUCTS, AND FLAME PROPAGATION

An analysis system capable of continuous measurement of CO, CO<sub>2</sub>, NO<sub>X</sub>, total hydrocarbons and oxygen (Ref. 3) during the burning of polymers was used in conjunction with smoke measurements. This system, designed and constructed at Monsanto Research Corporation (MRC), is connected to the NBS-Aminco smoke density chamber, that

is utilized for the burning of samples under controlled conditions. The apparatus and the test methods were described in some detail in a preceding quarterly report (Ref. 1). The test conditions were identical with those specified in that report.

Flame propagation measurements were conducted by the procedure specified for the ASTM Standard Method E 162-76. Either duplicate or triplicate measurements were performed with each material, depending upon the repeatability of results.

## 2.3 TENSILE STRENGTH AND ELONGATION MEASUREMENTS

An Instron Model TTC testing machine was used for the tensile strength and elongation measurements. It was equipped with serrated, Type 10F grips whose separation was 4.5 in. The specimens were cut with a Type I die. Their cross-sectional dimensions ranged from 0.041 in. x 0.500 in. to 0.073 in. x 0.500 in.

## 2.4 ELECTRICAL MEASUREMENTS

The volume resistivity was measured with a Rohde-Schwartz Company tera ohmmeter. The dielectric constant and dissipation factor measurements were conducted with a General Radio Company capacitance bridge, Type 716C. A Type 1690A sample holder was used for the latter deal measurements were conducted at 25

### 3. RESULTS AND DISCUSSION

The primary objective of the present program is to lower smoke formation from burning PVC and Neoprene polymer compositions. Concomitantly, enhancement of other fire performance characteristics (e.g., reduction of the rate of flame propagation) will be sought.

#### 3.1 FORMATION OF SMOKE

The advantageous effect of incorporated ferric and cupric acetylacetonates on the reduction of smoke from burning PVC was reported earlier (Ref. 1). Further reduction of smoke formation was sought with an alkyd- and an epoxy-based intumescent coating (Ref. 2). Both of these coatings reduced smoke formation from the base polymer that did not contain the smoke-retardant additives. However, when these coatings were applied onto the fire- and smoke-retardant compositions FSP-1 and FSP-2, they failed to improve the performance with regard to smoke formation (Ref. 2). The coatings themselves contributed smoke upon exposure to the radiant energy source and to the flame.

Two additional, more recently developed, fire protective coatings of different types were evaluated. These were a latex-type intumescent paint and a mastic coating. Measurements were conducted

with 1/16 in. and 1/4 in. thick samples. The results are listed in Table I. They are shown, together with the results for previously prepared compositions, in Figures 1-4.

It is interesting to note that the maximum smoke optical densities developed during the exposure of 1/4 in. thick samples are not much higher than the respective values for 1/16 in. thick samples (see Figure 1). The times required for the development of the maximum densities are longer with thick samples. The percentage char residue values are also higher with the thick samples (see Table II). The disproportionately low smoke optical density developed by thick samples is attributed to both physical and chemical factors. The char formed from the PVC resin is cellular, voluminous and insulating. Because of the larger mass, the thick samples are heated more slowly. Presumably, more extensive crosslinking occurs during the slower heating, causing a reduction of the amount of volatile (combustible and/or condensible) degradation products. Also, more extensive agglomeration of aerosol particles can occur during the prolonged degradation, causing an increase of light transmittance through the smoke.

Best performance was exhibited by smoke-retardant compositions coated with the alkyd-based intumescent paint, No. 110 by C. M. Athey Paint Company. As shown in Figures 2 and 3, all intumescent coatings, with the exception of the alkyd-based coating, caused an increase of the smoke optical density from the smoke-retardant compositions FSP-1 and FSP-2.

It should also be noted that the maximum smoke optical densities developed from the smoke-retardant compositions, coated with the epoxy- and latex-based intumescent paints, are on the average 24 percent lower than the value for the reference polymer of identical thickness.

The mastic coating (IC-3), applied in a greater thickness than the intumescent paints, contributed more to smoke than the latter. This is very evident from smoke optical density data obtained on coatings alone, applied onto aluminum foil supports (see Figure 4).

#### 3.2 FORMATION OF GASEOUS COMBUSTION PRODUCTS

The latex-based intumescent paint (IC-4) and the mastic (IC-3) reduced carbon monoxide formation under nonflame exposure conditions very significantly (see Table III and Figures 5 and 6). However, under flame exposure conditions, the maximum concentrations of carbon monoxide were not greatly affected by these coatings when they had been applied onto 1/16 in. thick specimens. In these respects, these two intumescent coatings functioned similarly to the alkyd- and epoxy-based paints tested earlier (Ref. 2).

Table I

SMOKE OPTICAL DATA FOR PVC COMPOSITIONS

	Thickness of Plastic	Maximum Smoke Optical Density		Time To Maximum Smoke Optical Density (min)	
Material	Sample (cm)	Flame Exposure	Nonflame Exposure	Flame Exposure	Nonflame Exposure
BP	0.16	510	320	4	10
FSP-1	0.64	240	260	17	22
FSP-2	0.64	230	195	20	20
FSP-1-IC-3	0.16	720	490	7	11
FSP-1-IC-4	0.16	340	260	7	9
FSP-2-IC-3	0.16	680	500	7	8
FSP-2-IC-4	0.16	310	240	7	9
FSP-1-IC-3	0.64	460	470	8	14
FSP-1-IC-4	0.64	320	250	15	16
FSP-2-IC-3	0.64	490	450	12	13
FSP-2-IC-4	0.64	330	260	13	20
A1-IC-3		490	330	10	16
A1-IC-4		140	93	19	17

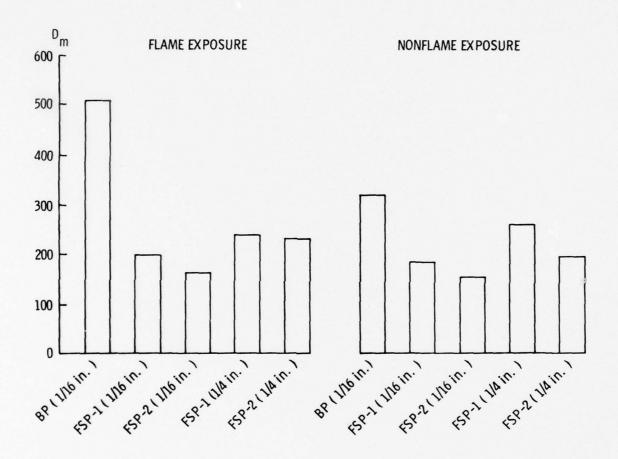


Figure 1. Maximum Specific Smoke Optical Densities of FSP Compositions

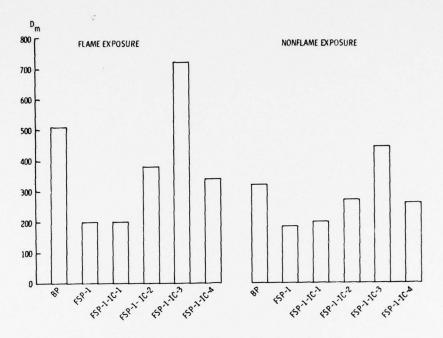


Figure 2. The Effect of Intumescent Coatings on Smoke Formation from 1/16 in. Thick Samples of FSP-1

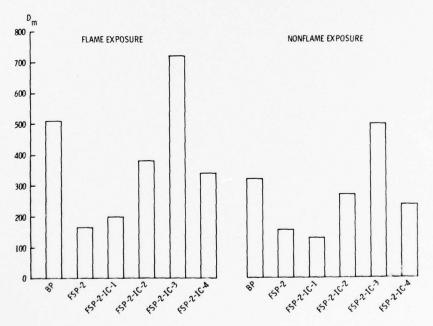


Figure 3. The Effect of Intumescent Coatings on Smoke Formation from 1/16 in. Thick Samples of FSP-2

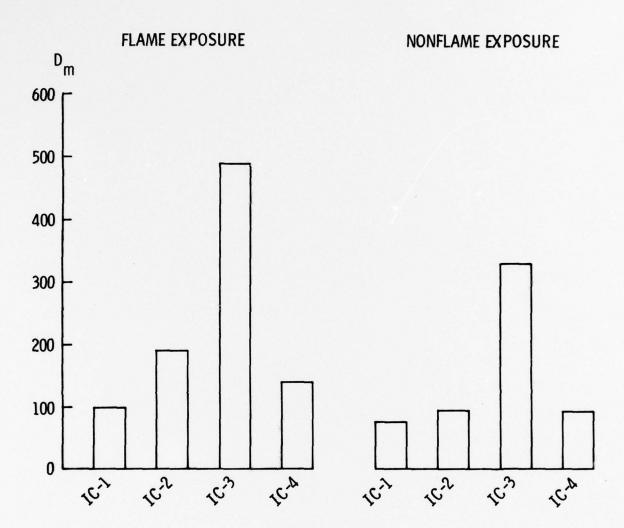


Figure 4. Maximum Smoke Optical Densities Generated from Intumescent Coatings, Applied Onto Aluminum Foil

Table II SAMPLE MASS DATA<sup>a</sup>

	Thickness of Plastic	Average Mass	Sample (g)	Average Mass	Consumed (g)
Material	Sample (cm)	Flame Exposure	Nonflame Exposure	Flame Exposure	Nonflame Exposure
FSP-1	0.64	45.4	46.4	23.5	22.2
FSP-2	0.64	35.0	33.0	21.4	15.6
FSP-1-IC-3	0.16	29.6	26.1	13.4	9.2
FSP-1-IC-4	0.16	21.3	18.7	10.6	7.4
EGD 2 TG 2	0.16	20.0	24. 2	12.0	0.0
FSP-2-IC-3 FSP-2-IC-4	0.16 0.16	28.9 17.6	24.2 16.2	12.9 9.2	9.0 7.2
FSP-1-IC-3	0.64	60.5	61.2	19.1	16.0
FSP-1-IC-4	0.64	39.3	47.1	18.4	12.1
FSP-2-IC-3	0.64	47.8	51.0	23.7	14.3
FSP-2-IC-4	0.64	42.3	40.6	13.0	10.5
A1-IC-3		28.4	31.2	8.4	6.5
A1-10-3		13.3	13.3	2.3	2.1
112 20 - 1		-5.5	± J• J	2.5	

 $<sup>\</sup>underline{\underline{a}}$  Sample length and width were 7.6 cm. The reported average values are based on either duplicate or triplicate experimental determinations.

Table III

MAXIMUM CO CONCENTRATION DATA FOR PVC COMPOSITIONS

	Thickness of Plastic	Maximum CO Concentration (ppm)		Time To Maximum CO Concentration (min)	
Material	Sample (cm)	Flame Exposure	Nonflame Exposure	Flame Exposure	Nonflame Exposure
ВР	0.16	1900	570	30	30
FSP-1	0.64	4800	1900	30	30
FSP-2	0.64	3300	2000	30	30
FSP-1-IC-3	0.16	1400	210	30	30
FSP-1-IC-4	0.16	2000	180	30	30
FSP-2-IC-3	0.16	2000	195	30	30
FSP-2-IC-4	0.16	1800	190	30	30
FSP-1-IC-3	0.64	1800	1150	30	30
FSP-1-IC-4	0.64	3100	580	30	30
FSP-2-IC-3	0.64	1250	290	30	30
FSP-2-IC-4	0.64	1000	240	30	30
A1-IC-3		1300	125	30	30
A1-IC-4		610	50	30	30

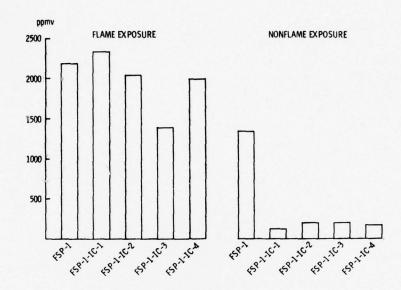


Figure 5. The Effect of Intumescent Coatings on Maximum CO Concentrations Developed from 1/16 in. Thick Samples of FSP-1

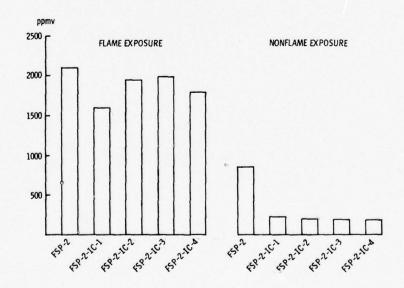


Figure 6. The Effect of Intumescent Coatings on Maximum CO Concentrations Developed from 1/16 in. Thick Samples of FSP-2

The latex-based intumescent paint and the mastic reduced carbon monoxide formation from the 1/4 in thick samples also under flame exposure conditions (see Figures 7 and 8). The average reduction was 65 percent from these thick samples.

It should be noted that carbon monoxide was generated under flame exposure conditions from all four coatings used on this work, when they had been applied onto aluminum substrate (see Table III). The formation of carbon monoxide from the coatings is attributed to the oxidation of the initially formed carbonaceous chars in the flame.

The formation of carbon dioxide from the FSP compositions was retarded by all intumescent coatings under the nonflame exposure conditions (see Table IV). The reduction of CO and  $\rm CO_2$  formation is an indication of the effectiveness of intumescent coatings for reducing the oxidation of the PVC compositions.

All intumescent coatings enhanced the formation of  $\mathrm{NO}_X$  under both types of exposure conditions; however, they delayed the times when the maximum concentrations were reached (see Table V and Figures 9-11). The alkyd-based coating made the smallest contribution to the nitrogen oxides among the gaseous degradation and combustion products. The formation of nitrogen oxides from the intumescent coatings is attributed mainly to the oxidation of the incorporated nitrogen-containing blowing agents.

Most intumescent coatings reduced the rates of <a href="https://www.nydrogen.chloride-evolution">hydrogen.chloride-evolution</a> and the maximum concentrations developed during sample exposures (see Table VI and Figure 12). The coatings were especially effective in reducing HCl formation from thick samples (see Figure 13). Apparently by retarding the rate of sample degradation, they cause the released HCl to have a longer residence time in the polymer matrix. Thereby, more time is available for reaction with the incorporated magnesium carbonate.

Intumescent coatings caused a modest increase of <u>hydrocarbons</u> evolution from the thin (1/16 in.) FSP compositions under flame exposure conditions (see Table VII and Figures 14-16). The reason is believed to be two-fold. First, hydrocarbons are generated from these coatings upon exposure to radiant heating and to a flame (see Figure 16). Secondly, the oxidation of the volatile organic degradation products from the FSP compositions to CO and CO<sub>2</sub> is retarded by the coatings.

With thick (1/4 in.) samples of FSP compositions, the mastic and the latex-based coatings retarded hydrocarbons formation by  $\sim\!20\%$  under flame exposure and  $\sim\!35\%$  under nonflame exposure conditions (see Table VII and Figures 17 and 18). The reduction of hydrocarbons formation from the thick specimens is attributed to significantly reduced pyrolysis.

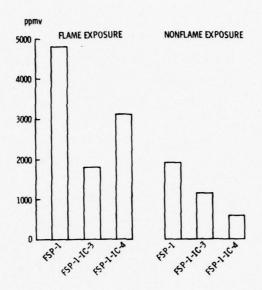


Figure 7. The Effect of Intumescent Coatings on Maximum CO Concentrations Developed from 1/4 in. Thick Samples of FSP-1

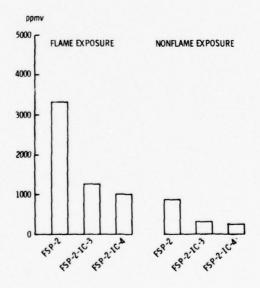


Figure 8. The Effect of Intumescent Coatings on Maximum CO Concentrations Developed from 1/4 in. Thick Samples of FSP-2

Table IV

MAXIMUM CO<sub>2</sub> CONCENTRATION DATA FOR PVC COMPOSITIONS

	Thickness of Plastic	Maximum CO <sub>2</sub> Concentration (%)		Time To Maximum CO <sub>2</sub> Concentration (min)	
_Material_	Sample (cm)	Flame Exposure	Nonflame Exposure	Flame Exposure	Nonflame Exposure
BP	0.16	1.78	0.12	30	30
FSP-1	0.64	2.70	0.89	30	30
FSP-2	0.64	2.36	0.72	30	30
FSP-1-IC-3	0.16	2.26	0.24	30	30
FSP-1-IC-4	0.16	1.59	0.16	30	30
FSP-2-IC-3	0.16	2.01	0.24	30	30
FSP-2-IC-4	0.16	1.78	0.15	30	30
FSP-1-IC-3	0.64	1.97	0.55	30	30
FSP-1-IC-4	0.64	1.58	0.35	30	30
FSP-2-IC-3	0.64	2.06	0.32	30	30
FSP-2-IC-4	0.64	1.80	0.24	30	30
A1-IC-3		1.86	0.24	30	30
A1-IC-4		1.14	0.06	30	30

Table V  ${\tt MAXIMUM\ NO_X\ CONCENTRATION\ DATA\ FOR\ PVC\ COMPOSITIONS}$ 

	Thickness of Plastic	Maximum NO <sub>X</sub> Concentration (ppm)		Time To Maximum NO <sub>X</sub> Concentration (min)	
Material	Sample (cm)	Flame Exposure	Nonflame Exposure	Flame Exposure	Nonflame Exposure
BP	0.16	19	4.6	5	4
FSP-1	0.64	5.4	2.6	30	30
FSP-2	0.64	7.8	2.4	30	30
FSP-1-IC-3	0.16	92	27	30	30
FSP-1-IC-4	0.16	56	20	30	30
FSP-2-IC-3	0.16	88	27	30	30
FSP-2-IC-4	0.16	56	18	30	30
FSP-1-IC-3	0.64	88	30	30	30
FSP-1-IC-4	0.64	37	18	30	30
FSP-2-IC-3	0.64	96	34	30	30
FSP-2-IC-4	0.64	61	21	30	30
A1-IC-3		82	44	30	30
A1-IC-4		53	32	30	30

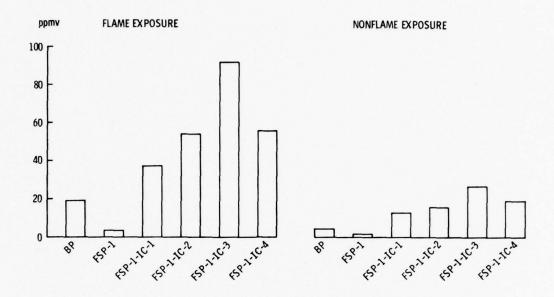


Figure 9. The Effect of Intumescent Coatings and Maximum  ${\rm NO}_{\rm X}$  Concentrations Developed from 1/16 in. Thick Samples of FSP-1

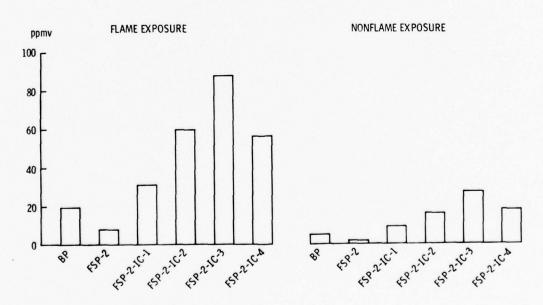


Figure 10. The Effect of Intumescent Coatings on Maximum  $\mathrm{NO}_{\mathrm{X}}$  Concentrations Developed from 1/16 in. Thick Samples of FSP-2

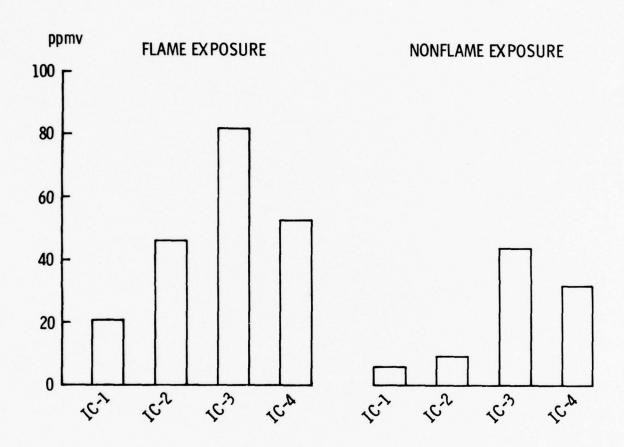


Figure 11. Maximum  $\mathrm{NO}_{\mathbf{X}}$  Concentrations Generated from Intumescent Coatings that Have Been Applied onto Aluminum Foil

Table VI

MAXIMUM HCl CONCENTRATION DATA FOR PVC COMPOSITIONS

	Thickness of Plastic	Concent			Time To Maximum HCl oncentration (min)	
Material	Sample (cm)	Flame Exposure	Nonflame Exposure	Flame Exposure	Nonflame Exposure	
BP	0.16	2400	2600	15	15	
FSP-1	0.64	7500	7800	30	30	
FSP-2	0.64	7300	3600	30	30	
FSP-1-IC-3	0.16	1200	1600	15	15	
FSP-1-IC-4	0.16	1400	2800	15	30	
FSP-2-IC-3	0.16	1100	1800	15	15	
FSP-2-IC-4	0.16	2900	3200	15	15	
FSP-1-IC-3	0.64	1900	2000	15	30	
FSP-1-IC-4	0.64	4000	2900	30	30	
FSP-2-IC-3	0.64	3700	3300	30	30	
FSP-2-IC-4	0.64	3700	2400	30	30	
A1-IC-3		580	530	30	15	
A1-IC-4		145	96	30	30	

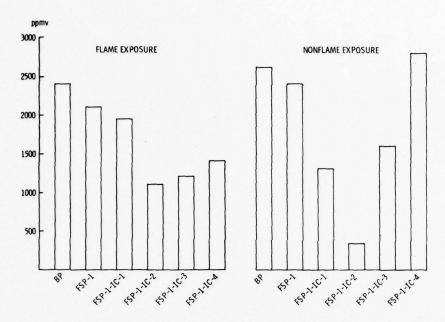


Figure 12. The Effect of Intumescent Coatings on Maximum HCl Concentrations Developed from 1/16 in. Thick Samples of FSP-1

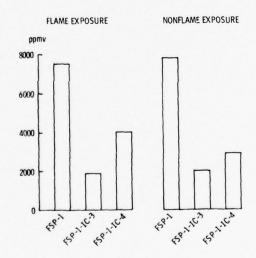


Figure 13. The Effect of Intumescent Coatings on Maximum HCl Concentrations Developed from 1/4 in. Thick Samples of FSP-1

Table VII

MAXIMUM HYDROCARBONS CONCENTRATION DATA FOR PVC COMPOSITIONS

	Thickness of Plastic	Maximum Hydrocarbons Concentration (ppm)		Time To Maximum Hydrocarbons Concentration (min)	
Material	Sample (cm)	Flame Exposure	Nonflame Exposure	Flame Exposure	Nonflame Exposure
ВР	0.16	5200	3000	30	17
FSP-1	0.64	9600	7300	30	30
FSP-2	0.64	9100	6800	30	30
FSP-1-IC-3	0.16	4300	3000	17	24
FSP-1-IC-4	0.16	4900	2700	30	12
FSP-2-IC-3	0.16	4600	3200	20	22
FSP-2-IC-4	0.16	4000	2700	30	22
FSP-1-IC-3	0.64	5600	5300	30	30
FSP-1-IC-4	0.64	8800	3900	30	30
FSP-2-IC-3	0.64	7400	5500	27	30
FSP-2-IC-4	0.64	8800	3900	30	30
A1-IC-3		3100	2400	25	30
A1-IC-4		2000	470	30	26

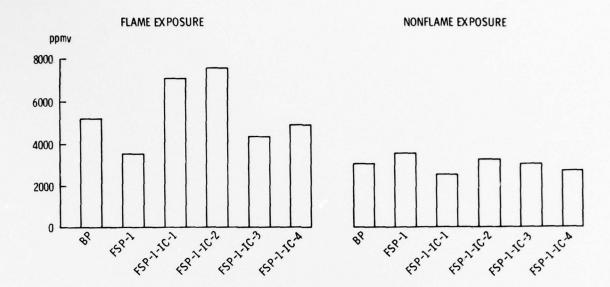


Figure 14. The Effect of Intumescent Coatings on Maximum Hydrocarbons Concentrations Developed from 1/16 in. Thick Samples of FSP-1

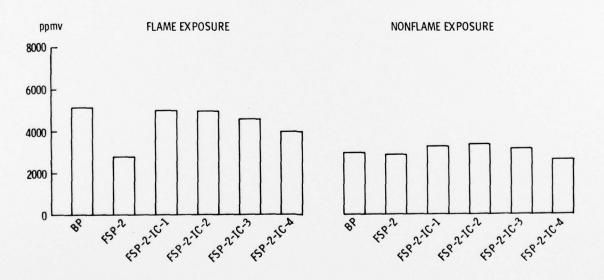
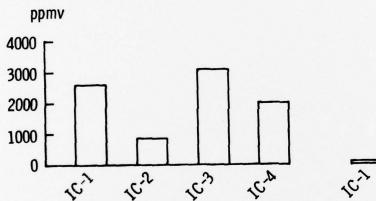


Figure 15. The Effect of Intumescent Coatings on Maximum Hydrocarbons Concentrations Developed from 1/16 in. Thick Samples of FSP-2

# FLAME EXPOSURE

# NONFLAME EXPOSURE



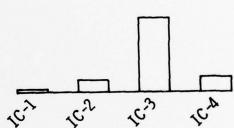


Figure 16. Maximum Hydrocarbons Concentrations Generated from Intumescent Coatings That Have Been Applied Onto Aluminum Foil

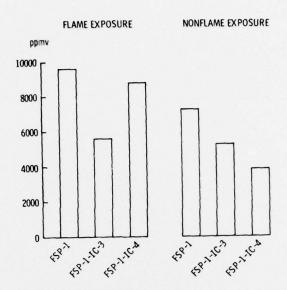


Figure 17. The Effect of Intumescent Coatings on Maximum Hydrocarbons Concentrations Developed from 1/4 in. Thick Samples of FSP-1

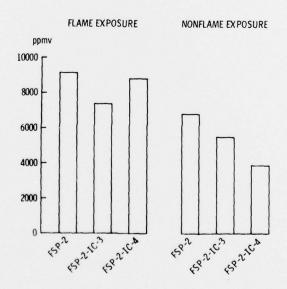


Figure 18. The Effect of Intumescent Coatings on Maximum Hydrocarbons Concentrations Developed from 1/4 in. Thick Samples of FSP-2

## 3.3 FLAME PROPAGATION

Vertically downward flame propagation rates for samples exposed to irradiative heating were measured by the ASTM Standard Test E 162-76. The data are summarized in Table VIII and the results for the individual tests are presented in Table XI in the Appendix. The mean distances of flame propagation for the different compositions, as functions of time, are presented in Figures 19 and 20.

According to this test (Ref. 4), the flame spread index,  $I_s$ , of a specimen is expressed as the product of the flame spread factor,  $F_s$ , and the heat evolution factor, Q.

$$I_S = F_SQ$$

The flame spread factor is defined in terms of the times in minutes  $(t_3, \ldots, t_{15})$  that transpire from the initial specimen exposure until the arrival of the flame front at the position  $3, \ldots, 15$  in. from the ignited sample edge.

$$F_S = 1 + \frac{1}{t_3} + \frac{1}{t_6 - t_3} + \frac{1}{t_9 - t_6} + \frac{1}{t_{12} - t_9} + \frac{1}{t_{15} - t_{13}}$$

The heat evolution factor is defined in terms of  $\Delta T$ , the maximum stack temperature rise in degrees Fahrenheit with reference to that observed with an asbestos-cement board specimen, and the experimentally determined constant  $\beta$ :

$$Q = 0.1 \frac{T}{B}$$

Physically, the constant  $\beta$  represents the maximum stack thermocouple rise in degrees Fahrenheit for unit heat input rate (btu/min) of the calibration burner. The factor 0.1 in the above equation is an arbitrary constant.

As indicated in Figures 19 and 20, the FSP compositions were found to propagate flames to greater distances than the reference composition, BP. The intumescent coatings applied onto the FSP-2 surface reduced the rate and distance of flame propagation. However, even the coated specimens propagated flames to a greater distance than the reference composition that did not contain magnesium carbonate and smoke-retardant transition metal-containing additives.

The following suggestion is offered as an explanation for the enhanced flame propagation characteristics of the FSP compositions. Because of the reaction of HCl with magnesium carbonate in the polymer matrix, the concentration of this flame-inhibiting species is reduced in the vapor phase. Therefore, the pyrolyzate formed from the FSP compositions is more flammable than the pyrolyzate formed from the reference composition. Consequently, the FSP pyrolyzate propagates flames more readily (i.e., at lower temperatures, and with greater dilution by air) than the pyrolyzate

Table VIII
FLAME PROPAGATION MEASUREMENT RESULTS

Material_BP	$\frac{F_s^{\underline{a},\underline{b}}}{(\min^{-1})}$ 2.7±0.2	$I_{s}^{\underline{b},\underline{c}}$ (btu x min <sup>-2</sup> ) $9.6\pm4.6$
FSP-1-IC-1	5.2±1.1	11.4±2.3
FSP-1-IC-2	9.6±1.0	21.6±8.9
FSP-1-IC-3	22.0±19.4	92.6±78.2
FSP-1-IC-4	5.7±0.1	12.5±0.2
FSP-2-IC-1	4.1±0.9	32.2±20.1
FSP-2-IC-2	6.5±1.8	33.2±7.1
FSP-2-IC-3	8.9±1.3	51.4±2.0
FSP-2-IC-4	3.9±1.3	15.7±7.2

 $<sup>\</sup>frac{a}{b}$ Flame spread factor, as defined in Ref. 4.  $\frac{b}{b}$ Mean value and standard deviation for duplicate or triplicate measurements.  $\frac{c}{b}$ Flame spread index, as defined in Ref. 4.

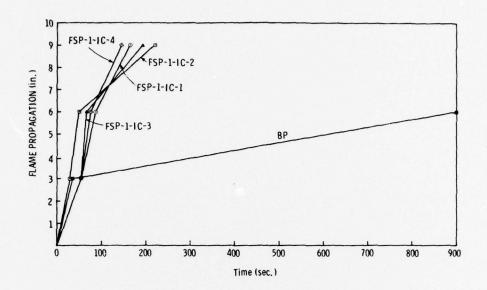


Figure 19. Flame Propagation by FSP-1 Compositions

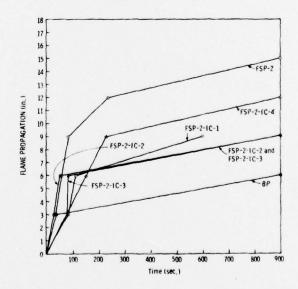


Figure 20. Flame Propagation by FSP-2 Compositions

formed from the reference composition under identical test conditions.

The following additional observations regarding flame propagation measurement results are of interest:

- Subsequent to the initial heating, most specimens propagated flames at a rapid rate from the 6 in. to the 9 in. distance from the point at which the samples were ignited. Thereafter, the rates of flame propagation became significantly slower.
- None of the samples propagated flames the entire length (18 in.).
- The flame spread factors  $(F_S)$  of the intumescent-coated copper-containing FSP-2 compositions were lower than those of the respective iron-containing compositions.

The compositions for which we sought to provide protection with intumescent coatings are already fire-resistant because of the chemical nature of the chlorine and phosphorus which they contain. Some reduction of the flame propagation rate was attained with the four intumescent coatings that were evaluated; however, more effective protection would be desirable.

Two approaches to the attainment of that goal merit evaluation. First, the intumescent coatings should be tested in thicknesses greater than those which have been found useful for the relatively more flammable wood substrates. Secondly, when needed for added protection of fire-resistant materials, the intumescent coatings should contain higher than the normally used concentrations of the fire-retardant ingredients.

## 3.4 TENSILE STRENGTH AND ELONGATION

The results of tensile strength and elongation measurements are summarized in Table IX. The mean values and the standard deviations are based on five determinations with each composition.

Table IX

# TENSILE STRENGTH AND ELONGATION DATA FOR SOME PVC COMPOSITIONS

Material	Yield Strength (psi)	Ultimate Strength (psi)	Elongation at Failure (%)
BP		3560±220	220±60
FSP-1	2930±70	3160±100	210±30
FSP-2	2930±100	2750±40	87±50

The base composition did not exhibit a yield point; the samples stretched until ultimately rupture occurred.

The ultimate tensile strength of the FSP-1 and FSP-2 compositions was 11 and 23 percent, respectively, lower than the value for the base polymer.

Whereas the value for elongation at failure for the FSP-1 composition was nearly identical with that for the base polymer, the samples of FSP-2 ruptured at lower elongation.

## 3.5 ELECTRICAL PROPERTIES

Volume resistivity, dielectric constant and dissipation factor measurements were conducted with the base polymer and with the two FSP compositions without intumescent coatings. The following results were obtained (Table X).

#### Table X

#### ELECTRICAL PROPERTIES

Material	Volume Resistivity (ohm-cm x 10 <sup>13</sup> )	Dielectric Constant	Dissipation Factor
BP	68	4.05	0.079
FSP-1	14	4.44	0.059
FSP-2	12	4.05	0.056

#### 4. ACKNOWLEDGMENTS

The authors are indebted to the following persons for assistance with various phases of the reported work: Mr. J. T. Miller for combustion products determinations, Messrs. A. W. Morgan, T. C. Mathis, and A. J. Balek for flame propagation measurements, Mr. C. D. Bias for electrical measurements, and Mr. D. L. Sheppard for tensile strength and elongation measurements.

### 5. REFERENCES

- 1. L. Parts and C. A. Thompson, "Flame- and Smoke-Retardant Polymer Systems," First Quarterly Report on Contract NOO024-76-C-5336, 15 September 1976.
- 2. L. Parts and C. A. Thompson, "Flame- and Smoke-Retardant Polymer Systems," Second Quarterly Report on Contract NOOO24-76-C-5336, 26 April 1977.

- 3. L. Parts, R. G. Olt and G. W. Wooten, "Products Resulting from Polymer Combustion," Polymer Conference Series, Flammability of Materials, University of Utah, Salt Lake City, Utah, July 11, 1974.
- 4. "Standard Method of Test for Surface Flammability of Materials Using a Radiant Energy Source," ASTM Designation E 162-76, Annual Book of ASTM Standards, Part 18, 1976.

APPENDIX

Table XI

EXPERIMENTAL FLAME PROPAGATION DATA

<u>Material</u> BP	t 3 <sup>a</sup> (sec) 38 32	t <sub>6</sub> a (sec) 861 866	t <u>9ª</u> (sec)		t <sub>15</sub> <u>a</u> (sec)	Δ <u>T</u> <u>b</u>
FSP-2	34	21	28	153	664	18
FSP-1-IC-1	54	45	58	741		8
	53	18	101	720		8
FSP-1-IC-2	36	20	172	247		13
	25	20	39	816		14
	23	21	304	552		7
FSP-1-IC-3	83	1.5	45	770		14
	41	4.1	81	344	429	22
	42	27	257	574		6
FSP-1-IC-4	60	21	78	738		8
	43	25	61	771		9
FSP-2-IC-1	62	42	197			50
	98	17	786			28
	46	54	43	757		10
FSP-2-IC-2	28	13	857			18
	26	32	841			20
FSP-2-IC-3	80	7.5	813			20
	67	9.8	823			23
FSP-2-IC-4	72	29	68	730		16
	83	115	87	614		13

 $<sup>\</sup>underline{a}_{t_3....t_{15}}$  represent times in seconds from initial sample exposure until the arrival of the flame front at the positions 3....15 in., respectively, along the length of the specimen.

 $<sup>\</sup>Delta T$  is the maximum temperature rise of the exhaust gases, in °C, with reference to the exhaust temperature measured with an asbestos-cement sample.

# DISTRIBUTION LIST

	No. of Copies
Naval Ship Engineering Center, Code N65197 National Center, Building No. 2 Room 5E52	2
Washington, D. C. 20362	
Naval Sea Systems Command, Code NOOO24 Department of the Navy Washington, DC 20360 Attn: NAVSEA 03511	1
Naval Surface Weapons Center, Code N60921 Silver Spring, Maryland 20910 Attn: Dr. Angel	1
Naval Research Laboratory, Code NOO173 Washington, DC 20390 Attn: Chemistry Div. Code 6100	1
Naval Research Laboratory, Code NOO173 Washington, DC 20390 Attn: Non-metallics Div.	1
Naval Ship Research and Development Center, Code NOO162 Bethesda, Maryland Attn: Non-metallics Code 284	1
Office of Naval Research, Code NOOO14 Ballston Tower #1, Code 472 Arlington, Virginia 22217	1
Naval Sea Systems Command, Code NOOO24 Department of the Navy Washington, DC 20360 Attn: NAVSEA 024	1
Defense Documentation Center, Code SL4200 Cameron Station Alexandria, Virginia 22314	12

UNCLASSIFIED
SECURITY CLASSIFICATION OF THIS PAGE (When Date Entered)

REPORT DOCUMENTATION	PAGE	READ INSTRUCTIONS BEFORE COMPLETING FORM
REPORT NUMBER	2. GOVT ACCESSION	NO. 3. RECIPIENTIS CATALOG NUMBER
. TITLE (and Subtitle)	and the Company of th	5. TYPE OF HEFORT & PERIOD COVERED
Flame- and Smoke-Retardant Polymer	Systems.	February - Aug 1977,
		6. PERFORMING ONG. REPORT NUMBER
. AUTHOR(s)		8. CONTRACT OR GRANT NUMBER(*)
Leo Parts, Robert D. Myers and Norr	man F./May	15 N00024-76-C-5336
Monsanto Research Corporation Dayton Laboratory, 1515 Nicholas Ro Dayton, Ohio 45407	oad	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
1. CONTROLLING OFFICE NAME AND ADDRESS		12. REPORT DATE
Department of the Navy, Code NO0024 Naval Sea Systems Command SEA 0253D		15 September 1977)
Washington, D. C. 20362		31 (2) 39/2).
4. MONITORING AGENCY NAME & ADDRESS(If differen DCASD, Dayton	t from Controlling Offi	Unclassified
Building 5, Code S3605A Dayton, Ohio		150. DECLASSIFICATION/DOWNGRADING
7. DISTRIBUTION STATEMENT (of the abstract entered	in Block 20, if differen	at from Report)
8. SUPPLEMENTARY NOTES		
KEY WORDS (Continue on reverse side if necessary an		
carbon dioxide flame pro carbon monoxide flame ret	ardant	nitrogen oxides polymers
combustion products hydrogen of the hydrogen o	chloride nt coating	polyvinyl chloride smoke
fire safety mastic		smoke retardant
ABSTRACT (Continue on reverse side if necessary and the effort directed to the enhancement of the polyvinyl chloride was contingent paint and a mastic coating on products were determined. Also, flowith these and with other, previous	ent of the fir nued. The eff the formation ame propagation ly prepared ex	re performance characteristics of fects of a latex-based intumes- of smoke and volatile combustion on measurements were conducted operimental compositions.
Among the protective coatings evaluated best performance with regard to contributed least to the NOx conten	o smoke and CO	) formation. This coating also
D 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOL	ETE	UNCLASSIFIED

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Date Entered)

20. Abstract (cont'd)

Tensile strength, dielectric constant, dissipation factor and volume resistivity data are reported for the experimental fire- and smoke-retardant PVC compositions.

UNCLASSIFIED